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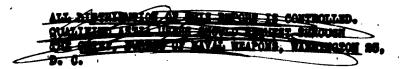
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INVESTIGATION OF GLASS-METAL COMPOSITE MATERIALS

NINTH QUARTERLY PROGRESS REPORT

COVERING PERIOD SEPTEMBER 15, 1957 to PECEMBER 15, 1957

CONTRACT NOrd 15764



by

HERBERT B AILES AND A. R. CANADY GLASS-METALS RESEARCH LABORATORY

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JANUARY 5, 1958

OWENS-CORNING FIRERGLAS CORPORATION Basic and Applied Research Center Newark, Ohio



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I. INTRODUCTION

The major efforts during the period covered by this report
(Sept. 15, 1957, to Dec. 15, 1957) have been in the following directions:

- A. Developing a glass-reinforced metal having utility in the 1500° 2000° F. range.
- B. Improving existing and investigating new methods of forming and working composites of aluminum and aluminum coated glass fibers.
- G. Determining a complete set of physical data on a standard glassreinforced aluminum composite in order to indicate the general characteristics of glass-reinforced metals as a class of materials.
- D. Developing a method of forming composites of aluminum and bare glass fibers and evaluating the physical properties of these composites.
- E. Production and testing of glass-reinforced aluminum tubular shapes.
- F. Developing a theory on the interaction of metals and glass fibers.

The work reported represents the combined efforts of Messrs. J. I. Aber,
R. E. Evans, B. B. Garick, P. A. Lockwood, H. E. Mathews, E. E. Mattern,
C. A. Riesbeck, E. W. Smart, R. S. Swain, G. E. Wince, and the authors
of the Glass-Metals Research Laboratory, and Dr. H. B. Whitehurst, Department Head. Acknowledgement is also made of the valuable assistance given
by many other members of the Basic and Applied Research Center.

Physical property measurements of the glass-reinforced metal test bars were performed by the Olin-Mathieson Chemical Corporation in New Haven,
Connecticut, and by the Ohio State Engineering Experiment Station of the
Ohio State University in Columbus, Ohio, under direction of Dr. T. S. Shevlin.

Work on fabrication of large shapes, in particular tubing, of glassreinforced metals was subcontracted to the Olin-Mathieson Chemical Corporation in New Haven, Connecticut. This subcontract was terminated on November 15, 1957, by direction of the Navy Bureau of Ordnance.

An extension of contract NOrd 15764 has been awarded by the Navy Bureau of Ordnance for one year from October 31, 1957, to October 31, 1958. Emphasis is to be placed on developing a glass-reinforced metal having utility in the 1500° - 2000° F. range.

This Ninth Quarterly Progress Report covers a transition period wherein the majority of the research effort is being redirected from glass-reinforced aluminum to higher temperature resistant glass-reinforced metals. Hence, this report also includes the completion of the Olin-Mathieson subcontract on production of large tubular shapes of glass-reinforced aluminum. In the future, research emphasis will be on a 1500°-2000°F, glass-reinforced metal with some work to be done on glass-reinforced aluminum to establish general characteristics of glass-reinforced metals and to allow investigation of techniques at lower operating temperatures.

II. SUMMARY

Copper is being used for preliminary investigation of methods of combining glass fibers and high melting point metals. Fibers can be coated at forming with a slurry of copper powder, dried, and then hot-pressed producing glass-reinforced copper. Unsuccessful attempts have been made to put more copper on the fibers by electrostatic precipitation and various spray techniques. A composite was made by shaking copper powder into glass fibers and subsequently sintering at 1600°F, under vacuum. A glass, X37B, with higher temperature resistance than E glass has been fiberised from a 50-hole bushing.

The variables involved in hot-pressing aluminum coated fibers have been evaluated and the optimum conditions have been established. Tensile strengths of 50,000 psi at room temperature can be consistently duplicated.

Room temperature repressing of composites to relieve residual thermal stresses between the glass and aluminum was unsuccessful.

Glass-reinforced aluminum has been tested by an aircraft component manufacturer as an actuator material operating at 500°F. For the specific operating requirements of this test, glass-reinforced aluminum was the only satisfactory material tested.

The electrical resistivity of aluminum is doubled by the addition of as little as 15% glass fibers. The resistivity is increased due to the volume of glass present and also due to the resultant smaller metal grain size.

A sample of hot-pressed aluminum coated fibers was suspended in a rocket blast at 5800°F. for 4-1/2 seconds without deformation. A short time tensile strength of 9300 psi was measured at 1090°F. on a standard composite of glass-reinforced aluminum. These tests indicate interesting properties of glass-reinforced aluminum for extremely high temperature, short-time applications.

Wetting studies indicate that titanium nitride is effective in increasing the wettability of glass fibers with molten aluminum.

Glass reinforced aluminum tubular shapes as large as 7" I.D. have been made successfully by centrifugal casting and by vacuum injection.

X-ray diffraction studies on various glass-reinforced aluminum composites show that the glass fibers tend to restrict grain growth and can have a grain refining action during casting and heat treating.

III. <u>DISCUSSION</u>

- A. DEVELOPING A GLASS-REINFORCED METAL HAVING UTILITY IN THE 1500 to 2000°F.

 RANGE
 - Selection of metal: The development of a glass-reinforced aluminum. has shown that by the addition of glass fibers to aluminum the utility of the metal can be increased to approximately 75% of its melting point. Using the 75% of the melting point criteria to get into the 1500 to 2000°F. range, the melting point of the metal would have to be in the 2000 to 3000°F. range. The metals in this temperature range which are being investigated are the alloys of copper, beryllium, mickel, iron and titanium (listed in the order of increasing melting point). The difficulty of forming the composites would increase approximately with the melting point of the metals. Since copper has the lower melting point, it is the logical metal for initial investigation towards the development of high temperature glass-reinforced metals. It is hoped that the knowledge gained from the formation of the copper composites could then be applied to the formation of composites with the higher melting metals.
 - 2. Selection of glass: The glass being used in all glass-reinforced aluminum composites at this time is a production borosilicate glass, designated as E glass. This glass was chosen because of its superior wetting properties with aluminum and because it retains a fair portion of its strength up to about 1100°F. In order to reinforce metals to have utility in the 1500 to 2000°F, range it is doubtful

that this glass would be satisfactory. It is difficult to form most of the higher temperature glasses on present equipment. It was felt that for the first investigation into the 1500 to 2000°F, range we should use a glass which could be easily formed on existing equipment and would give us the desired temperature range. To this end we have selected a new glass, X37B, which is easily formed on existing equipment and extends the useful temperature \$400°F, over E glass. Some of the other higher temperature glasses being investigated are fused silica, calcium aluminate glasses, and spodumene glasses. Fused silica fibers have been formed by leaching, by pulling from a molybdenum bushing protected with an inert atmosphere, and by direct flame melting of fused silica canes. These fibers have been coated with aluminum and lead with reasonable success. It is thought that the calcium aluminate and the spodumene glasses can be formed using the same techniques.

made by hot-pressing glass fibers coated with aluminum-copper alloys. It was hoped that these composites would have better tensile strengths at 1000°F, than the composites made by hot-pressing aluminum coated flass fibers. Composites were made by hot-pressing glass fibers coated with a 5% copper, balance aluminum, alloy. The glass fibers used to make these materials were not too satisfactory since the application of the copper-aluminum alloy resulted in a brittle coated fiber. Attempts to coat alloys with a higher copper content aluminum were impossible because of the brittle nature of the fiber

after it was coated. The results of the composites formed by hotpressing the 5% copper, balance aluminum, alloy coated fibers are shown in Table I. .

TABLE I

GLASS_REINFORCED 5≸ COPPER. BALANCE ALUMINUM ALLOY

Sample No.	Tensile Strength at 1000°F.
195	12,720
200	49,800
201	16,380

From this scant data, the first indications are that by increasing the copper content of the alloy, the strength is increased at elevated temperatures. However, these composites were very brittle and hard to handle.

Attempts were made to coat X37B fibers with various copper alloys.
65-35 brass was first used, but the fibers did not stand the high
temperature necessary. In an effort to get a low melting copper
alloy, a 10% magnesium, 90% copper alloy was formed which melted
at 1500°F. This temperature was also too high to coat the fibers.

In all cases the copper alloys used oxidised extremely, even with
a protective coating of powdered charcoal and various salts. Any
hot melt coating of copper alloys on glass fibers will have to include
a protective atmosphere.

Various copper alloys were vacuum injected into tubes containing bare glass fibers and aluminum coated glass fibers. Aluminum-

silicon brass, 65-35 brass, 10% magnesium and 90% copper alloy were injected in this manner. In all cases the glass fibers were badly damaged by the high temperatures involved. Higher temperature fibers will have to be used if the fibers are to be hot-coated or composites formed by any casting method.

X37B fibers have been coated at forming with a slurry of copper powder and ethylene glycol. These fibers are then placed in a die and heated to drive off the liquid vehicle. The fibers are then hot-pressed. Reasonable success has been achieved by forming copper-glass composites in this manner. Several samples have been made in this manner, but none have been tested.

Because of the detrimental effect on the fibers when they are hotcoated by higher melting metals, powder metallurgy techniques are being
explored to produce glass-reinforced higher temperature metals. The
two methods of forming these composites are by pressing at high
pressure and low temperature with subsequent sintering or by pressing
at low pressure and high temperature with no sintering operation. These
composites could then be hot extruded or rolled if desired.

Ultrasonic vibration is being investigated as a method of mixing the glass fibers and copper powder and shows much promise. Although copper is the material being used to formulate a method of combining the glass with higher temperature metals, other metals such as stainless steel, beryllium, and nickel are also being investigated coincident with the copper investigation.

Attempts are also being made to reinforce materials other than metals in order to develop a high temperature material. A sample of glass-reinforced ferric chloride has been prepared by crystallising the salt from concentrated solution onto a mass of fibers. The strength of the sample has not yet been evaluated. Other materials will be tried in the future including lithium fluoride because of its high lattice energy.

- B. IMPROVING EXISTING AND INVESTIGATING NEW METHODS OF FORMING AND WORKING COMPOSITES OF ALUMINUM AND ALUMINUM—COATED GLASS FIBERS
 - 1. Composites formed by hot-pressing aluminum-coated fibers
 - A previous report (1) showed that by hot-pressing aluminumcoated fibers at 600°F. and 34 tons per square inch, composites
 with tensile strengths as high as 47,920 per sould be formed.
 However, the results varied from 5,900 per to 47,920 per and it
 was deemed advisable to evaluate the hot pressing variables in
 order to get the maximum strengths possible. The highest
 temperature possible was used in all cases because it was found
 that at lower temperatures composites could not be formed
 satisfactorily regardless of the pressure. These specimens
 consist of 50-60% glass fibers in a relatively weak aluminum
 matrix. Table II gives a summary of the tensile strength data
 taken on these composites.

⁽¹⁾ H. B. Ailes - Seventh Quarterly Progress Report, Contract MOrd 15674

TABLE II
TENSILE STRENGTHS OF HOT-PRESSED ALUMINUM COATED GLASS FIBERS

Sample Number	Temperature Cf Forming,	And Time 'Of	Tensile Strength At Room Temp., psi	Average Tensile Strength, psi	Conclusions
120	700	5 min. pre- heat and 5 min. © 8.3TSI	27,200	27,600	
121			28,000		
156		5 min. pre- heat and 25	17,900		
157	670	sec. ● 8.3TS	I 9,320	13,000	
158	••		11,800		•
124		5 min. pre- heat, 1 min.	29,800		
125	670	● 8.3TSI, and 4 min. ● 16.0	19,950	30,700	
126		TSI	42,300		
127		5 min. pre- heat, 1 min.	52,400		
128	690	0 8.3 TSI, &		46,700	
129		4 min. ● 23.	42,600		23.4 TSI final pressure is adequate to form good Composites.
133		5 min. pre-	44,800		23.4 TSI as
134	670	heat, 1 min. 6 8.3 TSI, and		45,400	good as 30.1
135		4 min. 9 30.	47,900	•	

TABLE II (continued)

Sample Number	Temperature Of Forming,	And Time St	rength Room	Average Tensile Strength,	<u>Conclusions</u>
136		5 min. pre-	40,100		Initial press 0
137	670	heat, 5 sec. 0 8.3 TSI,	48,800	41,400	8.3 TSI helps some.
138		and 4 min. 6 30.1 TSI	35,400		
142	670	5 min. pre- heat, 10 min.	45,300	50,900	More time at initial press
143		● 8.3 TSI, & 5 min. ● 30.1 TSI	56,400		helps.
153		5 min. pre- heat, 15 min.	51,700		More time at final press helps.
154	670	0 8.3 TSI, & 1 min. 0 30.1	50,000	46,300	bress mether
155		TSI	37,100		
177		6 min. pre- heat, ‡ min. •	22,700		
178	630	2,4,6,8,16 TSI & 5 min. • 20.	, 31,000	22,500	
179		TSI	13,800		•
183		5 min. pre- heat, 2 min.	26,100		Stepwise appli- cation of pressure
184	650	● 2,4,6,8, 10,12,14, up to 32 TSI, and 5 min. ● 32 TS		38,400	probably does no good, but these results may reflect lower forming temp.

It appears that best composites are made by:

670°F. (or higher) die temp., preheat for 5 minutes, 10 minutes (or more) at \$.3 TSI and 4 minutes (or more) at 23.4 TSI.

It is quite possible that pressing at temperatures higher than 700°F. and at proportionally lower pressures would give stronger composites. No work has

been done in this area.

b. Repressing of composites to improve strength:

The tensile strength of glass-reinforced aluminum with 20-30% fibers and a 145-To matrix is approximately 30,000 psi at room temperature, while the compressive strength of the same material is approximately 130,000 psi. The large difference between tensile strength and compressive strength can possibly be explained by the prestressed state of the material. The coefficient of linear thermal expansion of aluminum is approximately four times as great as that of "E" glass. As the glass-aluminum composite cools from the casting temperature, the difference in the thermal expansion of glass and aluminum would cause the aluminum to be in tension and the glass to be in compression. When the composites are subsequently stressed in tension during tensile testing, the aluminum, already being in tension, may fail before the glass is able to assume a large part of the load. In compression the tensile stress in the aluminum is quickly relieved and the glass assumes a large part of the load. It might be possible to relieve the residual thermal stress by repressing at room temperature the composites to above the yield point of the aluminum. This might relieve the tensile stressed state of the aluminum, and the strength in tension would then theoretically be equal to that in compression. In an effort to prove this theory, hot-pressed composites were repressed at room temperature above the yield strength of the aluminum matrix. Table II gives a summary of the results of this repressing.

Table III

REPRESSED GLASS-REINFORCED ALUMINUM FORMED BY THE HOT-PRESSING TECHNIQUE

Sample Number	Hot Pressing <u>Conditions</u>	Tensile Strength At Room Temp., psi	Average Tensile Strength,	Percent Change in Tensile Strength
127	695°F. die temp. 5 min. preheat	52,400		-
128	1 min. • 8.3 TSI 4 min. • 23.4 TSI	45,200	46,700	
129	4 min. • 25.4 151	42.600		£27\$
130	Same as 127, 128, 129, except re-	52,900	54,600	
131	pressed cold at 23.4 TSI	57,000		
132		54,000		
142	670°F. die temp. 5 min. preheat 10 min. © 8.3 TSI	. 45,300	50,900	•
143		56,400		-6%
149	Same as 142,143,	50,200	47,900	
150	except repressed cold at 30.1 TSI	45,600		,
178	650°F. die temp.	31,000	22,500	
179	6 min. preheat 1 min. © 2,4,6,8, 16.6	13,800		•
	5 min. ● 20,TSI	٠.		4 65 ≴
180	Same as 178,179	56,300		
181	Except repressed cold at 33.5 TSI	25,800	36,800	
182		28,300	,	

When the optimum hot-pressing conditions are used, the recm temperature repressing does little good. When other than optimum conditions are used, the repressing probably does little more than improve the bonding of the composite and hence increase the tensile strength.

- DETERMINING A COMPLETE SET OF PHYSICAL DATA ON A STANDARD GLASS-REINFORCED ALUMINUM COMPOSITE IN ORDER TO INDICATE THE GENERAL CHARACTERISTICS OF GLASS-REINFORCED METALS AS A GLASS OF MATERIALS

 It was deemed advisable to get a relatively complete evaluation of the physical properties of a material which is essentially 145-T6 aluminum reinforced with 20-30 volume percent continuous longitudinally oriented glass fibers. The composite is made by the vacuum injection method. (1) This material was chosen as the best available 12 years ago when the evaluation began. Since that time, however, developments have been made which affect the physical properties of glass-metal composites. It has been decided to continue the evaluation of physical properties based on the original composite type so that a related set of physical property data will exist. This set of data can then be used to indicate the properties of materials which incorporate any future developments. The
 - 1. Modulus of rigidity and Poisson's ratio: The modulus of rigidity and Poisson's ratio have been measured by two different methods. The first method is by mounting strain gauges laterally as well as longitudinally on a tensile specimen, producing a ratio of the transverse contraction to longitudinal elongation. This ratio provides the basis for calculation of modulus of rigidity. The second set of values is obtained in torsion tests by measuring the angle of twist and by calculating the maximum shearing stress. The data is shown in Tables IV and V.

information collected during this reporting period follows:

⁽¹⁾ H. B. Whitehurst - First Annual Progress Report, Contract MOrd 15674

TABLE IV

MODULUS OF RIGIDITY BY POISSON'S RATIO

Test Number	Specimen Number	Strain Gages	n's <u>Ratio</u> Strain Gages 1 & 3 average	Modulus of Elasticity 10 psi	Modulus of Rigidity 10 psi
1	105-D	.114		10.2	4.578
2	105-D	.092		10.6	4.852
3	105-D	•090		10.2	4.679
1	105-D		.101	10.2	4.630
2	105 → D		•099	10.6	4.823
3	105-D		. 098	10.2	4.645
	Ave	rage .099		10.3	4.701

TABLE V

MODULUS OF RIGIDITY BY SHEARING STRESS AND ANGLE OF TWIST

Test <u>Number</u>	Specimen Number	Max. Shearing Stress	Angle of Twist Radians	Modulus of Rigidity
1	105B	19,072	.1201	3.253
6	105B	17,680	•0974	3.759
1	105F	16,710	.0670	4.215
3	105F	20.880	.94.00	4.872

4.025

Average

Poisson's ratio for unreinforced 148 aluminum is 0.33, while it is .099 for glass reinforced aluminum. The modulus of rigidity of unreinforced 148 aluminum is 4.0×10^6 psi, while it is 4.701×10^6 psi and 4.025×10^6 psi

- . 1. by the two different measurements for glass-reinforced aluminum. A possible explanation for the low value obtained for Poisson's ratio and the differences in the modulus of rigidity by the two measurements is that the glass-reinforced aluminum specimens used have continuous longitudinally oriented fibers and hence would have anisotropic properties.
 - 2. Electrical resistivity: Data on the electrical resistivity of the standard glass-reinforced aluminum composite made by the vacuum injection technique is shown in Table VI.

TABLE VI ELECTRICAL RESISTIVITY

Sample Number		Percent Glass	Resistivity (microhm-cm.)	Ay, Resistivity (microhm-cm.)
819-147 -	A	15%	6.42	
	В	15%	6.45	
	C.	15%	6.54	6.49
	ם	15%	6.43	
	7	15%	6.64	
819-146 -	A	19% .	6.66	
	D	19%	6.30	
	E	1%	6.29	6.47
	F	19%	6.64	
	G	19%	6.48	• •
819-145 -	В	22.5%	6.50	6.525
	D	22.5%	6.55	

TABLE VI(continued)
ELECTRICAL RESISTIVITY

Sample Number	Percent Glass	Resistivity (microhm-cm.)	Av. Resistivity (microhm-cm.)	
819-170 - A	30%	6.50		Samples
В	30%	6.45	6.42	Air-cooled Immediate-
C	30%	6.30		ly After Casting.
819 -148 - D	30%	5.74		Held at 900°F. for 3 hrs.,then slow-cooled in furnace.

The measured electrical resistivity of unreinforced 148 aluminum was 3.13 microhm-cm. The addition of as small as 15% glass more than doubles the electrical resistivity. Increasing the percentage glass from 15% to 30% had little effect on the resistivity. It is thought that the resistivity of the composites is increased by the glass cross section and also by the refinement in the aluminum grain structure due to the glass. The one 30% glass composite held at 900°7. for a considerable length of time and then slow-cooled had a much lower resistivity than the 30% glass composites that were cooled in air immediately after casting, showing that the smaller grain size definitely increases the resistivity.

3. Compressive strength: Previous measurements of the compressive modulus of elasticity of glass-reinforced aluminum indicated that the modulus was in the order of 5 x 10⁶ psi. This very low value was obtained by using the pross-head travel of the testing unit to calculate the modulus; this method is quite questionable because

3. it reflects end effects of the specimen. More reliable compressive tests have been made using a Baldwin-Lima-Hamilton Strain Gage System to measure strain; these measurements are not affected by end effects of the specimens. This data is shown in Table VII.

TABLE VII

COMPRESSION TESTING OF GLASS-REINFORCED ALUMINUM

Sample Number	Ultimate Compressive Strength (psi)	Ultimate Compression	Compressive Yield Strength (0.2% offset)	Compressive Modulus, psi
1	(1) 89,000	5.2%	41,500	9.9 x 10 ⁶
3	(2)	(2)	56,500	10.7 x 10 ⁶
7	131,000	5.0%	52,700	10.7 x 10 ⁶

⁽¹⁾ Sixteen previous ultimate compressive strength tests indicated an average of 128,000 psi with a low of 101,000 psi.

The above tests were conducted on samples approximately .375 inches in diameter and .750 inches in length. They were tested at a strain rate of .01 inches per minute. Ultimate compressive strength was calculated as the ratio of breaking load to original cross-sectional area.

This data indicates an average compressive yield strength of 50,200 psi at room temperature and an average compressive modulus of 10.4 x 10^6 psi at room temperature. Unreinforced 145 aluminum has a compressive yield strength of 60,000 psi at room temperature and a compressive modulus of 10.7 x 10^6 psi at room temperature.

⁽²⁾ Test not carried to failure.

Wear resistance: A previous report (1) indicated that the wear resistance of the standard glass-reinforced aluminum composites was poor. Tests made in a Falex Wear Tester showed that the glass-reinforced aluminum specimens had considerably less wear resistance than either the unreinforced aluminum or the standard steel specimen.

Recent tests of glass-reinforced aluminum as an actuator material operating at 500°F. have shown that by hard anodising the standard glass-reinforced aluminum composites the wear resistance is excellent. Several aluminum alloys in the configuration of female splines were run against mil-5-6758 steel male splines to determine load-carrying ability, wear, gall characteristics, and coefficient of friction at average spline pressures of from 775 to 2,000 pei, at 500°F. with various lubricants. A hard anodized standard glass-reinforced aluminum composite in the form of female splines lubricated with Texas Uni-temp 500 grease performed far superior to all other materials tested. This hard anodised standard glass-reinforced aluminum composite as a female spline mated with a mil-S-6758 male spline withstood 40,726 cycles at 2,000 psi average spline pressure at 500°F. ambient temperature with no galling or seising and very little wear. The requirements for the test were 20,000 cycles at 1,000 psi average spline pressure.

⁽¹⁾ H. B. Ailes - Seventh Quarterly Progress Report Contract MOrd 15764

- 5. Short-time, high-temperature properties: A sample of hot-pressed aluminum coated glass fibers was suspended in a recket blast at 5800°F. for 42 seconds without deformation. No comparative results for other materials are available, but three seconds is considered average for most metals. A 20-30 second tensile strength of 9300 psi was measured at 1090°F. on a standard composite of glass-reinforced aluminum. These tests would indicate some interesting properties and possible applications of glass-reinforced aluminum at very high temperatures for very short times.
- D. DEVELOPING A METHOD OF FORMING COMPOSITES OF ALUMINUM AND BARE GLASS FIBERS AND EVALUATING THE PHYSICAL PROPERTIES OF THESE COMPOSITES

 Wetting studies: A number of surface active agents were investigated by CMCC to determine their effect on wettability of "E" glass. The results of this study are included in Appendix A.
- E. PRODUCTION AND TESTING OF GLASS-REINFORCED ALUMINUM TUBULAR SHAPES

 It was found desirable in the best interest of this project to subcontract to the Olin-Mathieson Chemical Corporation work on fabricating
 large glass-reinforced aluminum tubes. The CMCC subcontract has been
 terminated by direction of the Navy Bureau of Ordnance, effective
 November 15, 1957. A detailed description of the conclusion of this
 work is included in Appendix A.
- P. DEVELOPING A THEORY ON THE INTERACTION OF METALS AND GLASS FIBERS

 The combination of most metals and glass fibers results in some degradation in the strength of the glass fibers. It is extremely important to understand the interaction which takes place between the

metal and glass fibers. Once the interaction is more clearly defined, it will, in all likelihood, be possible to control this interaction and produce composite materials of glass and metals having the most desirable properties. Previous studies (1) do not definitely indicate whether the fiber strength degradation is due to chemical, thermal, or mechanical reaction of the metal upon the fiber. Attempts could be made to isolate the three degradation effects and evaluate each separately. The chemical effect would be isolated by coating the fibers with a silicone prior to metal coating. The thermal effect would be isolated by coating the fibers by various chemical methods and by vacuum coating methods which deposit metal on the fiber at comparatively low temperatures. The mechanical effect would be studied by analysis of half coated fibers where the degradation due to mechanical means is minimized.

1. X-Ray diffraction studies: A series of glass-reinforced aluminum composites and suitable metal control samples were x-rayed to study the resultant diffraction patterns. By studying the patterns of the samples in the "as cast" condition and after various heat treatments, it is possible to estimate the effect of glass fibers on the grain growth of the metal. Note that following the specified heat treatment the samples were cooled in air. The data on these studies is shown in Table VIII.

⁽¹⁾ H. B. Ailes - Eighth Quarterly Progress Report, Contract NOrd 15764

TABLE VIII

GRAIN SIZE (larger number indicates larger grains)

	As Cast	After 2 hrs. 9 300°F.	After 2 hrs. 9 600°F.	After 2 hrs. e 900°F.
14 S Aluminum	7	7	7	9
Glass-Reinforced Aluminum (Vacuum Injected)	6	7	6	4
Glass-Reinforced Aluminum (Hot-Pressed Aluminum Coated Glass Fibers)		1	1	2

The samples formed heavy oxide coatings during high temperature heat treatments and this may have clouded the results somewhat. The glass fibers tend to restrict grain growth and can have a grain refining action during casting and heat treating. The amount of restriction and refinement varies with the percent glass present and production method.

IV. FUTURE WORK

This contract has been renewed for one year from October 31, 1957, to October 31, 1958. The work during this period will be confined to developing a glass-reinforced metal having utility in the 1500-2000°F. range. Work will be continued on the phases of glass-reinforced aluminum that will have a direct relation to higher temperature composites.

The subcontract to Olin-Methieson to produce glass-reinforced aluminum tubular shapes by centrifugal casting and by vacuum injection was discontinued on November 15, 1957.

Various uncoated refractory fibers will be combined with Gu, Ni, S.S., Be and Ti powders and hot-pressed in order to evaluate the most promising combinations. Tensile tests of these combinations at 1500-2000°F. will be used to select the best metal and the best glass. Subsequent work will be towards improving methods of combining this glass and this metal.

Attempts will be made to vacuum inject various brasses and bronses into tubes containing the new X37B fibers. The new fibers will also be coated with some of the copper alloys under a protective atmosphere to get an indication of single fiber strengths.

X37B fibers will be vacuum injected with aluminum and tested at 1000°7. for an indication of the high temperature properties of this glass.

Glass-reinforced aluminum will be isostatically repressed in an effort to relieve the severe thermal stress in the composites and realise the maximum strength possible. Physical testing of glass-reinforced aluminum composites will continue.

Impact strength, notch sensitivity, stress-rupture characteristics,
fatigue strength, wear resistance, and corrosion resistance will be
measured at temperatures up to 1000°F. The water absorptive capacity
and wet strength of glass-reinforced aluminum will be measured. These
measurements are intended to indicate the general characteristics of glassreinforced metals.

V. APPENDIX A

PRODUCTION OF LARGE GLASS REINFORCED ALUMINUM CYLINDERS
COVERING PERIOD SEPTEMBER 15, 1957 TO NOVEMBER 15, 1957

SUBCONTRACT UNDER NOrd 15764

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WARREN H. KAYE
METAL-GLASS RESEARCH SECTION
GLASS, METALS AND PLASTICS DEPARTMENT

DECEMBER 30, 1957

OLIN MATHIESON CHEMICAL CORPORATION Research Laboratories New Haven, Connecticut

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I. INTRODUCTION

Olin Mathieson Chemical Corporation (CMCC) has been a subcontractor to Owens-Corning Fiberglas Corporation (CCF) under contract NOrd 15764 for the purpose of producing and testing large glass-reinforced aluminum cylinders, and making physical property measurements of experimental CCF glass-reinforced aluminum samples. The contract reads as follows:

"Work under this subcontract shall be devoted to fabrication of large shapes, particularly tubing, of glass-reinforced aluminum and testing of glass-metals samples. It is understood that CMCC has available the knowledge and facilities for metallurgical process development. Considerable heavy-duty metal-working equipment can be made available."

The subcontract was issued effective November 1, 1956, through November 1, 1957. The first report covered activities from inception of the program through September 15, 1957.

Subsequent to that date, NOrd requested OCF to rewrite their proposal for contract renewal to cover investigation of reinforced metals having utility at 1500-2000°F. Since basic research to develop new ceramic fibers and methods of combination with higher melting metals than aluminum would comprise the major effort for at least the first year of the new program, it was not contemplated that any work would be subcontracted by OCF during this phase. It was, therefore, provided in the new prime contract that sufficient funds would be allocated to bring the OMCC work on glass-reinforced aluminum cylinders to a rapid conclusion. OMCC estimated a cut-off date of November 15, 1957, which was met.

This constitutes a final report covering the period September 15 through November 15, 1957. The major efforts during this period have in general conformed to the program of Future Work outlined in the previous reports

- A. Further development of centrifugal casting techniques.
- B. Product evaluation.
- C. Vacuum impregnation of Fiberglas forms.
- D. Vacuum injection of cylindrical shapes.
- E. Treatment of fibers to enhance wettability.
- F. Physical testing, OCF samples.

The work reported represents the combined efforts of the personnel of the Metal-Glass Section of the Glass, Metals and Plastics Department headed by Mr. Meade McArdle.

II. SUMMARY

The 7.7 inch diameter centrifugal casting machine described in the previous report (1) was further modified with a dual drive arrangement to permit casting at low rpm. Refinements include installation of a direct reading tachometer and a limit switch on trough travel. The machine was completely realigned, bearing surfaces polished, and the total assembly was dynamically balanced. Product quality was improved by closer control over rpm and pouring rate, use of an improved vacuum dried Fiberglas preform, preheating of the mold, and casting of heavy wall tubes. A total of 31 centrifugal castings was made during this period. Optimum preform characteristics and casting procedures have been delineated on page 32 of this report.

An apparatus was built to test the bursting strength of several centrifugally cast glass-reinforced aluminum tubes. Empirical tensile tests of ring sections cut from centrifugal castings yielded room temperature data which compared favorably with results for the cast allow with no glass reinforcement.

Construction of the vacuum impregnation apparatus was completed and several trial runs were made. Preliminary results were encouraging but experiments were discontinued so that the vacuum pump could be used with the vacuum injection process for production of glass reinforced cylinders.

The fourth attempt to produce a glass-reinforced aluminum cylinder by the vacuum injection technique resulted in a satisfactory tube which was tested for hoop strength at room temperature.

⁽¹⁾ W. H. Kaye, Appendix, 8th Quarterly Progress Report, Contract NOrd 15764

It was found that of a number of chemicals investigated, titanium nitride is apparently most effective in promoting penetration and wetting of Fiberglas preforms with molten aluminum. Time did not permit evaluation of this material in centrifugal casting experiments.

The physical testing of various COF samples was continued, including more accurate determinations of compressive strength and compressive modulus.

III. DISCUSSION

A. Development of Centrifugal Casting Techniques

1. Mold speed and casting rate

Earlier experiments indicated that shear forces developed in high speed casting of molten aluminum into Fiberglas preforms tended to tear the fibers and distort the preform geometry, while the impact of the metal compressed the fibers before full penetration could be achieved. It was believed that better glassreinforced aluminum castings could be obtained by minimizing these forces while the metal was being cast and then rapidly accelerating the mold to effect maximum penetration. Accordingly, an auxiliary low speed drive was installed on the centrifugal casting machine and a direct reading tachometer coupled to the flask. Buns #78 through #89 were made with a 6 inch iron pipe insert and sand liner in the 7.7 nch mold to accommodate 6 inch diameter Fiberglas preforms left over from earlier experiments. Runs #90 through #107, with the exception of #93, #96, #97 and #100 which incorporated no glass, were made with an improved 7.7 inch preform wound by CCF to specifications outlined in the last report(1). Detailed experimental data are recorded in Table I.

The minimum rpm required to prevent raining of metal in the 7.7 inch flask was 400 rpm. The casting rate becomes more critical at lower speeds and best results were obtained at rates of 1/2 lb./sec. or lower. This procedure largely eliminated the undesirable effects

⁽¹⁾ W. H. Kaye, Appendix, 8th Quarterly Progress Report - Contract MOrd 15764

of shear and compression on the preform, and more uniform metal-glass distribution resulted. However, penetration was not significantly improved because the lag in acceleration to high speeds due to transmission losses permitted the metal to freeze before maximum centrifugal force was developed. Somewhat better results were obtained by slow-casting at a steady 1400-1600 rpm throughout. See casting #95, Figure I.

2. Wall thickness

To obtain satisfactory metal penetration over the range of mold speeds investigated, it was necessary to cast twice as much metal as theoretically required to form a cylinder of uniform composition. This had the effect of multiplying the radial pressure developed in the moltan aluminum without resorting to higher speeds with attendant shear and impact effects. However, where a 1/4-inch wall Fiberglass preform is used, composite cylinders of about 1/2-inch wall are produced, and the excess aluminum on the inner wall must be bored out to produce a finished tube of uniform metal-glass composition. Compare in Figure I the end view of casting #95 with the end of casting #47 which has been turned and bored to 0.250 inch wall.

3. Improved preform, vacuum dried at 600°F.

The previous report noted certain deficiencies in the Fiberglas preform structure and recommended incorporation and standardisation of important characteristics. OCF succeeded in making preforms

which met the principal requirements of uniformity in overall dimensions, integrity, fiber type, etc., and which were much easier to process. A vacuum drying chamber was built to accommodate these preforms as well as the tube windings later made for vacuum injection forming of glass-reinforced aluminum cylinders. After several hours of drying under 3 mm vacuum at 600° F., the preform would be quickly inserted in the 7.7-inch mold which was also heated to 600-700°F. immediately prior to casting. In this way, the effects of variations in adsorbed moisture were eliminated and casting temperatures standardized. It was expected that better penetration and fiber wetting would result under these conditions, although these effects could not be observed directly.

4. Optimum casting conditions

The best 7.7-inch glass-reinforced aluminum castings from the standpoint of physical appearance, surface characteristics, uniformity of composition and wall thickness, were obtained under the following or similar conditions, which may be considered optimum for this particular glass fiber-aluminum and alloy system: (See casting #95, Figure I.)

Preform: Bare fiber, 1 mil, 1 end, wound on and affixed to expanded aluminum screen to 0.D. of 7.7-inch, wall 1/4 inch, glass weight 1 lb., vacuum dried at 600°F., temperature 600°F. at casting.

Metal: Aluminum alloy (355) SC51A, fluxed with Goveral 70, weight 19 lb. (approximately double shot), temperature 1350°F.st casting, rate of pour 1/3-1/2 lb./second uniformly rear to front of mold.

Mold: Permanent, stainless steel, 7.7-inch I.D., temperature 600°F. at casting, RPM constant at 1500.

B. Product Evaluation

1. Physical appearance, composition

Glass-reinforced aluminum castings made during this period are evaluated in Table II on the basis of overall quality including surface characteristics, uniformity of composition and wall thickness, penetration, etc.

2. Bursting strength tests

Apparatus was designed and constructed for hydraulic testing of glass-reinforced aluminum cylinders for bursting strength. Castings #42, #47, and #73 made prior to this period (1) were machined into suitable specimens, burst under hydraulic pressure at room temperature and hoop strengths calculated. Although no elevated temperature tests were made, experience indicates that room temperature strengths would be maintained up to 700-800°F.

Cylinder	I.D.(inches)	Wall (Reduced Section)	Hoop Strength (psi)
#42	7.149	.149	11,950
#47	7.358	.110	16,720
#73	6.860	.250	15,100

⁽¹⁾ W. H. Kaye, Appendix, 8th Quarterly Progress Report, Contract Nord 15764

Figure I shows test specimens made from castings #47 and #73.

Figure II shows a test in progress, cylinder #42 in the test fixture and two views of the above cylinders after testing. The tubes were found to be quite porous under hydraulic pressure, and it was necessary to seal them with an application of sodium silicate.

3. Tensile tests of ring sections

To save machining time associated with preparation of bursting strength specimens, ring sections were cut from cylinders #86, #91, #93 and #100 and stressed to failure in hoop tension with special test fixtures made from half-round segments of the same radius.

Some stress concentration is induced in the sample by this method, and hoop strengths calculated from bursting tests would undoubtedly be higher. It is believed that this procedure is suitable for comparative purposes, however, and is quicker and easier to perform.

Cast No.	Hoop Strength, psi (room temperature)								
86	Bare glass, 550°F., 6.5 inch O.D.,	1,482							
	sand liner, steel insert at 300°7.,	1,650							
	350-3200 rpm	2.070 Av. 1,734 psi							
91	Bare glass, 600°F., 7.7 inch O.D.,	15,000							
	Stainless steel mold at 650°F.,	9.375							
	1400 rpm	Av. 12,187 psi							
93	355 allow only, 7.7 inch 0.D.,	17,000							
	stainless steel mold at 650°F.,	13,800							
.:	350-1400 rpm	Av. 15,400 pei							

Cast No.	Hoop Strength, psi (room temperature)									
100 ,	355 allow only, 7.7-inch O.D.,	23,250								
•	stainless steel mold at room	20,750								
	temperature, 1500 rpm	24,250								
		24.100								

Av. 23,087 pmi

Test results for the 355 aluminum alloy castings are included for comparison with glass-reinforced aluminum specimens, and in the case of #100 are a little more than 2/3 the published figures for tensile strength of permanent mold castings of 355 alloy, as cast.

The very low values for easting #86 are not explainable.

C. Vacuum Impregnation

The vacuum impregnation apparatus was completely assembled and tested satisfactorily under applied vacuum and pressure. Some difficulty was encountered with burn-out of the heater elements in melting operations. However, a trial run was made under vacuum only, using a dish-shaped preform of flexible filter mat. The bottom sup parted from the holder assembly when the glass fiber mat was compressed, and interrupted the experiment. Nevertheless, a 2-inch diameter section at the bottom of the preform showed an encouraging degree of metal penetration. The preform holder was reinforced and further experiments were scheduled to develop procedures and techniques and evaluate the effect of pressurising the unit with nitrogen.

However, it was necessary to discontinue experiments with this equipment so that the auxiliary vacuum pumping equipment could be used in the

investigation of an alternate method of producing glass-reinforced aluminum cylinders by vacuum injection techniques as described in the previous report. (1)

D. Vacuum Injection of Glass-Reinforced Aluminum Tubes

The apparatus and procedure have been described earlier (1) except that flanged tube connections were substituted for welds to facilitate removal of the glass-reinforced aluminum tube. Two aluminum-coated Fiberglas windings were made on OMCC mandrels in the Newark OCF labs with approximately 8% glass loading; two were made in the OCF Ashton Plant with approximately 16% glass loading. The tube assemblies were dried under vacuum at 600°F., preheated rapidly in an electric furnace to 1000°F., and the open end inserted into the molten aluminum with application of 3mm vacuum at the opposite end of the assembly. The first three runs resulted in incompletely formed cylinders, but the fourth yielded a well formed glass-reinforced aluminum tube of uniform metal-glass composition and wall thickness, 17 inches long x 5 inches I.D., 5-1/2 inches O.D. This was considered a successful demonstration of the feasibility of this method for producing tubes of limited length. Ring sections were out from sound areas of each tube and tested as described above, with results comparable to those obtained with centrifugal castings.

⁽¹⁾ W. H. Kaye - Appendix, 8th Quarterly Progress Report-Contract MOrd 15674

Winding	Ring Section	Hoop Strength, psi
#1 Newark	1 inch from bottom end as injected	11,530
	2/4 inches from bottom	10,330
•	4/5 inches from bottom	14.300
		Av. 12,053 pel
#2 Newark	1 inch from bottom	13,700
	2 inches from bottom	16,350
	4/5 inches from bottom	16.000
		Av. 15,350 pei
#1 Ashton	Bottom	12,300
	2/4 inches from bottom	13,800
	4/5 inches from bottom	14.300
		Av. 13,466 pml
#2 Ashton	1 inch from bottom	16,750
		Av. 16,750 pei

The above figures indicate greater strength in sections farther from the end submerged in molten aluminum. It may be assumed that the fibers in these sections have not suffered as much strength degradation due to thermal effects and/or chemical reaction with molten aluminum.

E. Fiber Coatings

Laboratory investigations were made to find surface active agents or other treatments of OCF "E" glass fiber materials which would 1) enhance wettability of the "E" glass fibers by molten 35% alloy, 2) act as a fiber spacer to assist penetration by the metal, and 3) temporarily rigidise the preform to facilitate handling, impart dimensional stability and eliminate longitudinal contraction in the epinning mold.

Certain plastic coatings appeared promising based on lab results, and an attempt was made to evaluate two of these (polystyrene and polymethylmethacrylate) in centrifugal casting runs #47, #51 and #59 (1). The bursting strength results obtained recently with easting #47 (polystyrene soaked preform) compare favorably, above, with these obtained for #42 (1 mil bare fiber preform) and #73 (2 mil, 1/2 aluminum-coated fiber preform) and indicate that polystyrene or similar plastics may be found useful in accomplishing the three desired objectives.

A number of surface active agents such as titanium, vanadium, columbium and tantalum carbides and nitrides were investigated in the laboratory for their possible effect on wettability of "E" glass fibers with 355 aluminum alloy. Recent studies indicate that titanium nitride apparently enhances wettability when dispersed in finely divided form through a Fiberglas bundle. Objective 3 is not met by this system, and the surface activity of this material has not been evaluated in centrifugal casting operations.

F. Physical Testing. OCF Samples

During this period, room temperature physical properties of 42 glassreinforced aluminum samples submitted by OCF have been determined in
the OMCC laboratories. These tests have included determination of
Tensile strength, elongation, modulus of elasticity in tension, ultimate
compressive strength and modulus of elasticity in compression. Test
results were reported directly to OCF.

⁽¹⁾ W. H. Kaye, Appendix, 8th Quarterly Progress Report, Contract NOrd 15764

IV. FUTURE WORK

The CMCC subcontract under NOrd 15764 was terminated by agreement between CCF-CMCC effective November 15, 1957, and no further work is contemplated.

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8

Product Evaluation

Metal rained from the top of the flask during low speed casting, disrupting the preform.

However, considerable metal drained into annular space between Raining eliminated by increasing rpm. insert and flask, Drainage eliminated by using sand liner between insert and flask. Inner surface of casting rough, small amount of glass exposed. Relatively poor penetration, variation in wall thickness.

Inner surface unimproved. Penetration fairly good. Wall thickness not uniform. 8

Some metal-coated fibers exposed on inner casting surface. Penetration Wall thickness more uniform. fairly good. 8

3 Uneven wall thickness. Poor penetration.

84 Preform collapsed. Pouring rate too high.

Wall thickness uneven. Relatively poor penetration. Small areas glass exposed, inner surface. 42

85 Statlar to 844.

Some areas of bare glass on inner surface. Improved penetration.

7 Random areas bare glass inner surface.

Menetration good.

Strip of bare glass evident on inner casting surface. Good penetration otherwise.

Good penetration. Mall thickness uniform. One small area of bare glass at inner surface.

From wall thickness. Ridged metal-coated interior. Penetration fair.

2 Good penetration. Uneven wall thickness. Good inner surface.

Uneven wall thickness. Outer surface pitted. Aluminum alloy 355 casting only, go glass incorporated. Interior good.

Uneven wall thickness. Areas of bare and metal-coated fibers on inner wall. Penetration poor.

(See Figure 1). Small area of bare glass outer wall. Good penetration. X

Aluminum alloy 355 only. Poor surface appearance.

Exterior less pitted than #96. Aluminum alloy 355 only.

Marrow, longitudinal strip bare glass on inner surface. Penetration good otherwise.

Similar to #98. Uneven wall thickness. Some bare glass areas on inner wall. Penetration good.

Aluminum alloy 355 only. Good surface appearance, even wall thickness. 8

Similar to #99. ğ

Similar to #99, except penetration not as good. 201

Penetration fair. Smooth inner surface. 19.

Good penetration. g

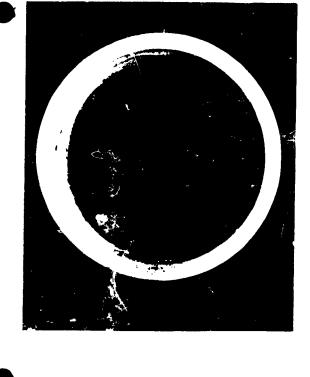
Similar to #104. ä Similar to floh. 200 Similar to #104, except small section bare glass inner surface. B

Average dimensions as cast: 5 1/2 inches I.D. x 6 1/2 inches O.D. x 3000 gms. Thirteen glass-reinforced aluminum centrifugal castings were made with a 6 inch iron pipe insert and sand liner in the 7.7 inch mold. 10 inches long. Average weight: FOTES:

Average weight: 7500 gms. Fourteen glass-reinforced aluminum cylinders were cast in the 7-7 inch stainless steel permanent mold. Average dimensions: 6 3/4 inches I.D. x 7 1/4 inches O.D. x 17 1/2 inches long. Average weight: 750

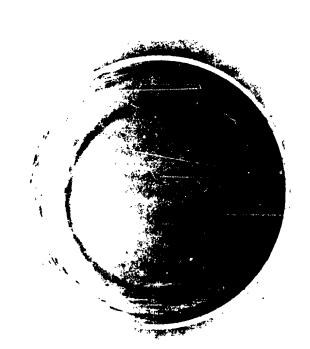
Average dimensions: Four aluminum (no glass incorporated) cylinders were cast directly in the flask. Average weights 5600 gas. 7 inches I.D. x 7 3/4 inches O.D. x 17 inches long.

CENTRIFUGAL CASTING #73 AFTER MACHINING FOR TEST.



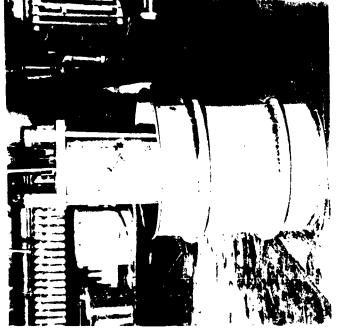
CENTRIFUGAL CASTING #95, END VIEW.

CENTRI FUGAL CASTING #95 AS CAST.

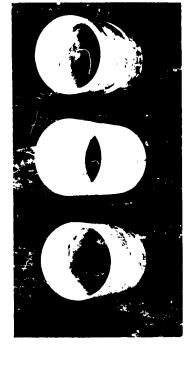


CENTRIFUGAL CASTING #47, After Machining for test.





CENTRIFIGAL CASTING #47 IN BURSTING STRENGTH TEST FIXTURE.



CENTRI FUGAL CASTING SPECIMENS AFTER BUFSTING. 14 £42 £73



111 #12 £73

	770-		porgl	A.S						0.37 25	30.0	*
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80	61	••	2	**	160	**	11	**	3632	1800 10	inter	
81	64	••	**	**	164	**	**	**	3650	1300		
82	46	**	1	**	175	•	**	••	3630	1450	4	
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90	125	••	"	1	380	600	19-3/4 hr.at 600°1 in vacuum tank	P .,	8172	# P		2/5
91	127	**	**	17	510	600	"	**	**	•		1/4
92	126	**	*1	**	600	750	2-3/4 hr Inside Flask	••	••	;;	.,,	1/2
93	None							11	11	1350	1	
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95	128	***	••	11	475	700	In side F lask	*1	8172	1300	* "	
96	None			 '				11	4994	11		1
97	None					-		19	5902	1350		3/5
98	131	Bare	1	1	410	650	Inside Flask	**	8172	1400	1	1/3
99	138	1/2A1	,,	"	460		19 hr. at 600°F	1.	"		centor	1-1/5
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102	140	"	"	-	480	"	3 hr. "	•	"	••		•
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All custing done with end pour trough.

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**	1350				**	п	**	650	17	**	7315
6356	10	•	i ve	1/3	**	**	11	700	1450	1450	5760
8172	1300	- !			11	**	11	**	1580	1580	7920
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8172	1400	`	1 4	1/3	**	**	Gas Flame on Outside	650	400	**	8070
	1300	in.	cont or	1-1/5	31	**	Od CB13C	600	400	3000	8480
4994	1350		*		**	**	None	Room	1500	1500	4982
8172	"		Į.,,	1	**	**	Gus Flame on Outside	600	400	3000	8275
0112	•,	•	I in		**	**	None	Room	150 0	**	8465
1.9	**	Pet	-Front	3/5	17	**	Gas Flame on Outside	650	**	**	7690
	,,				11	**	None	Room	••	1500	8385
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4086 5 4086											
Gas			£				•				



